

A COORDINATED FOCUSED ION BEAM/ULTRAMICROTOMY TECHNIQUE FOR SERIAL SECTIONING OF HAYABUSA PARTICLES AND OTHER RETURNED SAMPLES. E.L. Berger¹ and L.P. Keller², ¹GeoControl Systems – Jacobs JETS Contract, NASA Johnson Space Center, Mail Code JE23, Houston, TX 77058, USA (eve.l.berger@nasa.gov). ²NASA Johnson Space Center, Mail Code KR, Houston TX 77058.

Introduction: Recent sample return missions, such as NASA's Stardust mission to comet 81P/Wild 2 and JAXA's Hayabusa mission to asteroid 25143 Itokawa, have returned particulate samples (typically ~5-50 μm) that pose tremendous challenges to coordinated analysis using a variety of nano- and micro-beam techniques. The ability to glean maximal information from individual particles has become increasingly important and depends critically on how the samples are prepared for analysis. This also holds true for other extraterrestrial materials, including interplanetary dust particles, micrometeorites and lunar regolith grains.

Traditionally, particulate samples have been prepared using microtomy techniques (e.g., [1]). However, for hard mineral particles $\geq 20 \mu\text{m}$, microtome thin sections are compromised by severe chatter and sample loss. For these difficult samples, we have developed a hybrid technique that combines traditional ultramicrotomy with focused ion beam (FIB) techniques, allowing for the *in situ* investigation of grain surfaces and interiors. Using this method, we have increased the number of FIB-SEM prepared sections that can be recovered from a particle with dimensions on the order of tens of μm s. These sections can be subsequently analyzed using a variety of electron beam techniques. Here, we demonstrate this sample preparation technique on individual lunar regolith grains in order to study their space-weathered surfaces. We plan to extend these efforts to analyses of individual Hayabusa samples.

Instrumentation: Microtome thin sections (~50-70nm thick) were prepared on a Leica EM UC6 ultramicrotome with a diamond knife. Additional electron-transparent thin sections were prepared using an FEI Quanta 600 3D dual beam FIB-SEM. TEM analyses were carried out on the JEOL 2500SE field-emission scanning transmission electron microscope (STEM). All instruments are housed at NASA JSC.

Embedding & Microtomy: A ~40×40×20 μm lunar dust grain from highland soil 64501 was embedded in low viscosity epoxy. The particle was partly sectioned to a depth of ~10 μm ; sections were placed on Cu grids with thin amorphous films for TEM analyses. At this stage the potted butt is available for SEM, microprobe, and other analyses.

With the sample surface exposed, the epoxy bullet was trimmed to a height of ~0.5cm (fig. 1) to accommodate the allowable dimensions for FIB work. Using a diamond trim knife on the microtome, the

epoxy surrounding the grain was removed on 3 sides (to within 1-2 μm of the sample), leaving the sample at the end of a rectangular box sitting above the surface of the bulk of the epoxy. The depth of material removed extended ~15 μm below the bottom of the particle. The trimmed sample was attached to an SEM pin mount using carbon tape, and the sides of the epoxy bullet were coated with conductive paint. The entire assembly was coated with ~40nm of carbon, to eliminate sample charging during FIB work (fig. 2).

FIB-SEM & TEM: A protective carbon cap was placed according to the plan for the 14 FIB sections (fig. 3). For this grain, the cap was applied in steps, to determine minimum distances required between sections. For future sections, the entire cap will be completed prior to any milling. The central 'spine' of the cap runs perpendicular to the front of the sample, and the 'ribs' protruding from either side run parallel. Each rib indicates the location of a planned FIB section, and the spine contains the final two planned sections. We used a cap with a 4 μm wide spine and 2 μm wide ribs that have $\geq 3\mu\text{m}$ of space between them (narrower cuts resulted in too much re-deposition of material inside the trenches).

Using a 30kV, 3nA ion-beam we exposed the front surface of the grain and commenced milling the trenches on side 1. Fig. 4 shows an oblique view of the first 4 trenches, after initial ion-milling at 52° and further thinning at 50.5° and 53.5°. Rather than using the typical C-cut to prepare the sample for lift-out, an L-cut is used instead, leaving the sample connected by an interior tab. Each section on the right was removed, starting at the front of the grain. The procedure was repeated on the left-hand side of the grain, leaving a spine of material from which the last 2 sections are made (fig. 5). Once lifted out, the sections are attached to TEM grids and thinned to electron transparency using traditional FIB techniques (e.g., [2]).

Our hybrid technique preserves both interior and edge features, including delicate features such as amorphous silicate rims and solar flare tracks (fig 6), and allows for a more thorough characterization of μm -scale particles than either FIB or ultramicrotomy would afford alone.

References: [1] Bradley J.P. and Brownlee D.E. (1986) *Science*, 231, 1542-1544. [2] Zega T.J. *et al.* (2007) *MAPS*, 42, 1373-1386.

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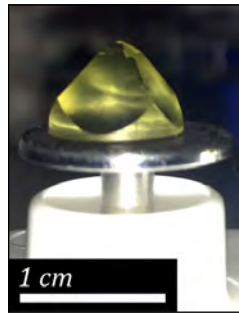


Figure 1. Trimmed epoxy bullet attached to an SEM pin mount.

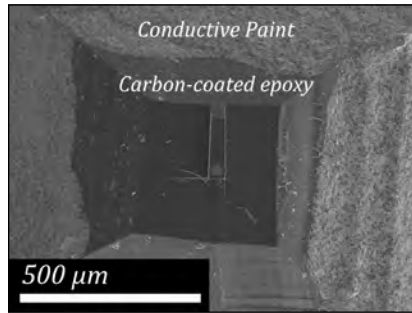


Figure 2. Secondary electron image (SEI) of the top of the grain mount, after coating with conductive paint and carbon.

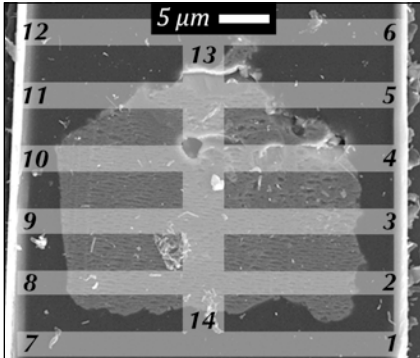


Figure 3. SEI of planned protective carbon cap placement. Sections 1-12 run horizontally along the ribs, sections 13 and 14 run vertically along the spine. The numbering is on the outside edges of the planned sections.

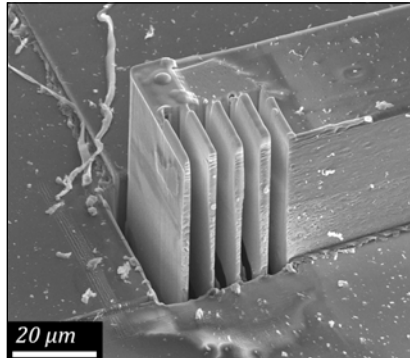


Figure 4. SEI showing an oblique view of sections 1 through 4, with material milled away from either side, prior to milling the L-cuts and lifting the sections out.

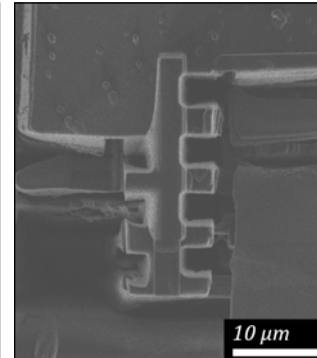


Figure 5. Ion beam image of the mount after sections 1, 2, 3, 4, 7, 8, & 9 were lifted out.

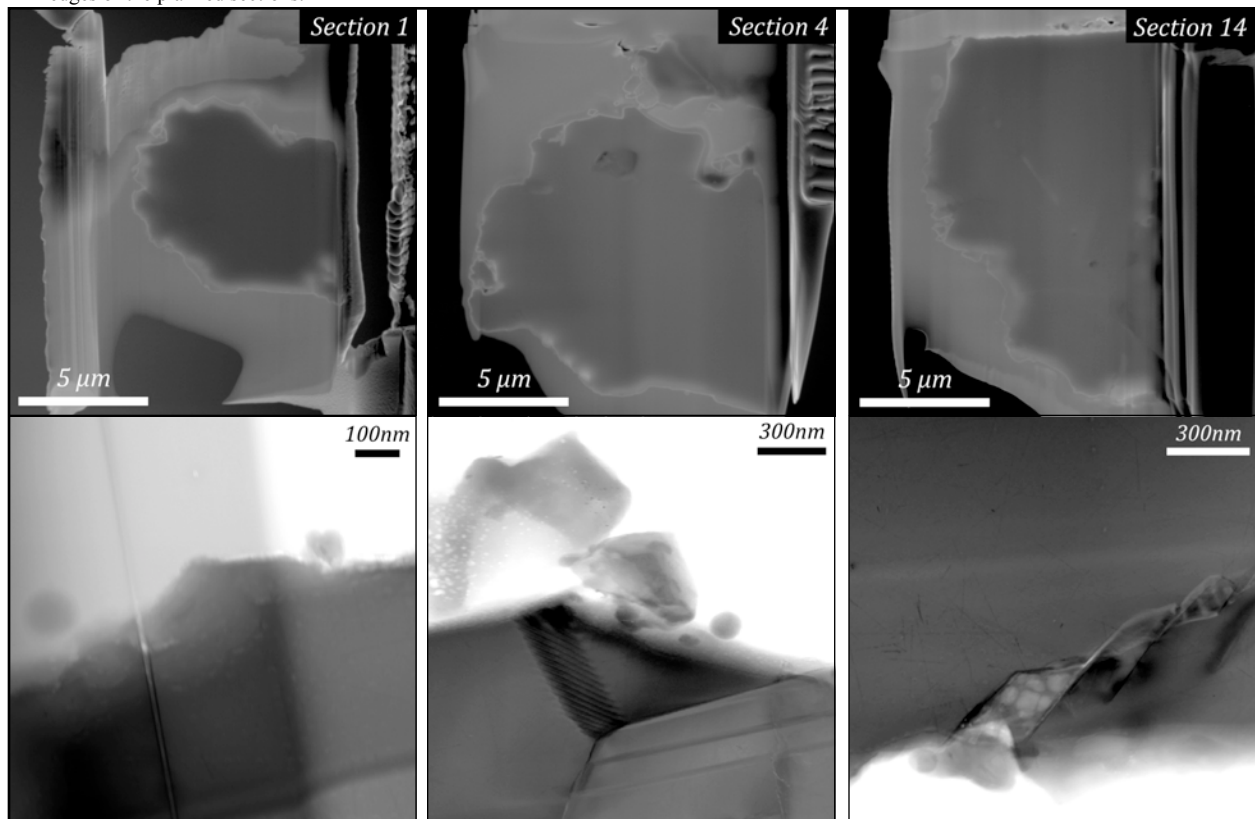


Figure 6. The top 3 images are SEIs of sections 1, 4, and 14. The particles are surrounded by epoxy, with the carbon caps on top and TEM grid bars to the right. Below each SEI is a TEM bright field image (BFI) of a portion of the edge of each section. Features of space weathering (solar flare particle tracks, disordered rims, and nanophase Fe particles decorating the outer edges) can be seen in the BFIs.